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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

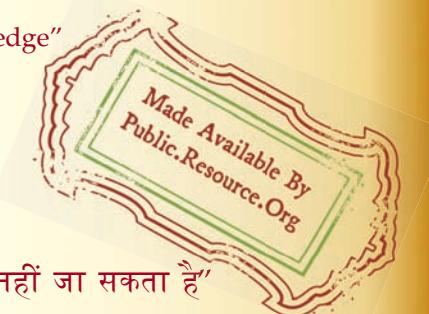
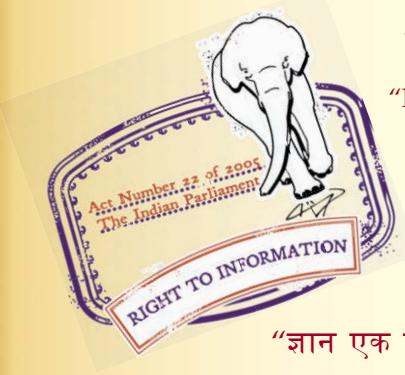
“Step Out From the Old to the New”

IS 504-1 to 12 (2002): Chemical Analysis of Aluminium and its Alloys -- Parts 1 to 12 [MTD 28: Methods of Chemical Analysis of Non-Ferrous Metals]

“ज्ञान से एक नये भारत का निर्माण”

Satyanaaranay Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक
अल्युमिनियम और उसके मिश्रधातुओं
का रासायनिक विश्लेषण
भाग 1 से 12
(दूसरा पुनरीक्षण)

Indian Standard
CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS
PARTS 1 TO 12
(*Second Revision*)

ICS 77.120.10

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard (Parts 1 to 12) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical/Instrumental Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1954 and subsequently revised in 1963. This revision of the standard covers following parts:

Part 1	Determination of Silicon and Lead
Part 2	Determination of Iron
Part 3	Determination of Copper
Part 4	Determination of Zinc
Part 5	Determination of Manganese
Part 6	Determination of Magnesium
Part 7	Determination of Nickel
Part 8	Determination of Chromium
Part 9	Determination of Tin
Part 10	Determination of Antimony
Part 11	Determination of Titanium
Part 12	Determination of Vanadium

The main changes in this revision from its earlier version are as below:

- Part 1 The spectrophotometric method for determination of silicon up to 0.1 percent has been incorporated and determination of lead by the sulphide method has been updated.
- Part 4 Determination of zinc by dithizone photometric method and by 'Mercuric thiocyanate method' have been updated.
- Part 5 Determination of manganese in the range of 0.01 to 0.10 percent by periodate (photometric) method and up to 0.5 percent by persulphate method have been deleted.
- Part 6 EDTA method for determination of magnesium in the range 0.5 to 11 percent has been deleted.
- Part 8 Determination of chromium by persulphate oxidation method has been incorporated.
- Part 11 Spectrophotometric method for determination of titanium in the range 0.001 to 1.0 percent using diantripyrilmethane has been incorporated as an alternate method.
- Part 12 Determination of vanadium in the range 0.005 to 0.02 percent using N- cinnamoyl- N- phenyl- hydroxamic acid (spectrophotometric) method has been incorporated as new part which was not covered in the standard earlier.

The standard although revised in parts is being printed in as a single publication for the convenience of users.

Parts 13 to 16 covering Determination of Sodium, Berillium, Zirconium and Lithium are under preparation and shall be printed as soon as finalized.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

*Indian Standard***CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS****PART 1 DETERMINATION OF SILICON AND LEAD***(Second Revision)***1 SCOPE**

1.1 This standard (Part 1) describes the following methods for determination of different ranges of silicon in aluminium and its alloys.

<i>Range (Percent by mass)</i>	<i>Method</i>
Up to 0.10	Spectrophotometric method
0.02-0.03	Molybdenum yellow (photometric method)
0.3 and above	Gravimetric method

1.2 It also describes the method for determination of lead by the sulphide colorimetric method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
266 : 1993	Sulphuric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

**5 DETERMINATION OF SILICON IN
ALUMINIUM METAL BY SPECTRO-
PHOTOMETRIC METHOD****5.1 Outline of the Method**

The silicon in the sample solution is converted to silicomolybdate complex, and subsequently reduced by ascorbic acid. The spectrophotometric measurement is made at 815 nm.

5.2 Reagents**5.2.1 Sodium Hydroxide Solution (15 percent)**

Dissolve 15 g of sodium hydroxide pellets in 100 ml distilled water. Store in a plastic container.

5.2.2 Dilute Nitric Acid (BN)

To 493 ml of distilled water slowly add 507 ml of nitric acid and stir to mix completely.

5.2.3 Ammonium Molybdate Solution (14.5 percent)

Dissolve 14.5 g of ammonium molybdate in 100 ml water and store in a plastic container.

5.2.4 Tartaric Acid Solution (10 percent)

Dissolve 10 g of tartaric acid in 100 ml distilled water and store in a plastic container.

5.2.5 Dilute Sulphuric Acid (16 N)

To 566 ml of distilled water, slowly add 434 ml of sulphuric acid with constant stirring and cooling under tap water.

5.2.6 Ascorbic Acid Solution (2 percent)

Take 2 g of ascorbic acid and dissolve it in 100 ml distilled water. Store in a plastic container. Always use a freshly prepared solution.

5.2.7 Aluminium Solution for Matrix Correction

Take 1 000 g aluminium wire of purity 99.9 percent minimum, and dissolve in 20 ml of sodium hydroxide and extract the content in 50 ml of nitric acid (8 N). Make up the volume to 250 ml. Store in a plastic container.

5.2.8 Standard Silicon Solution A (100 mg/l)

Fuse 0.214 g of anhydrous silica with 2 g of sodium carbonate in platinum crucible. Cool the melt, dissolve completely in water and dilute to 1 000 ml in a volumetric flask. Store the solution in a polyethylene bottle.

5.2.9 Standard Silicon Solution B (10 mg/l)

Transfer 10 ml of solution A (5.2.8) to a 100 ml volumetric flask and make up the volume with distilled water.

5.3 Procedure

5.3.1 Weigh accurately 1 000 g of sample and transfer to a cleaned nickel crucible or teflon/stainless steel beaker of 100 ml capacity. Add 20 ml of sodium hydroxide (15 percent) and cover the crucible by a lid with a slight opening. Wait for 5 to 10 min and open the lid. If some of the portion remain undissolved then heat slowly to dissolve all.

5.3.2 Carefully pour the contents to a beaker containing 50 ml of nitric acid (8 N) and transfer the washings of the crucible with hot water in to the same beaker. Heat slowly over a hot plate until all the salts get dissolved. Cool, transfer the solution to a 250 ml Polyethylene volumetric flask with the washings of the beaker with distilled water, and make up to the mark with distilled water.

5.3.3 Take 10 ml of sample solution in 100 ml beaker.

5.3.4 Add 10 ml of ammonium molybdate solution (5.2.3). Wait for 10 min to complete the reaction. Check the pH of the solution and bring it to 0.85-0.90 by dropwise addition of dilute nitric acid. Transfer the solution to a 100 ml volumetric flask with the washings of the beaker with distilled water. To it add 5 ml of tartaric acid and shake gently for mixing. Add 11 ml of sulphuric acid (16N). Cool the solution under tap water to 25-30°C. Add 2 ml of freshly prepared ascorbic acid solution and make up the volume with distilled water. Note the time. Measure the absorbance/concentration at 815 nm after 10 min but not beyond 30 min.

5.3.5 Find the concentration of the sample solution from the calibration curve and calculate the percentage of silicon as given in 5.4.

5.3.5.1 Calibration curve

Take six No. 100-ml beaker and use one of them as blank. To each of it add 10 ml of aluminium solution for matrix correction. Serially add 1, 2, 3, 4 and 5 ml of standard silicon solution B (5.2.9) and follow rest of the procedure as specified in 5.3.4.

5.3.5.2 Draw the calibration curve by plotting the concentration of the 5 standard solutions of silicon vs absorbance.

5.4 Calculation

$$\text{Silicon, percent by mass} = \frac{M \times 250}{m}$$

where

M = mass, in mg/ml of the sample found in aliquot taken from the calibration curve, and

m = mass, in g of the sample taken.

6 DETERMINATION OF SILICON BY THE MOLYBDENUM YELLOW SPECTRO-PHOTOMETRIC METHOD (FOR SILICON CONTENT BETWEEN 0.02 TO 0.3 PERCENT)**6.1 Outline of the Method**

The sample is dissolved in sodium hydroxide and the solution acidified with nitric acid. The orthosilicic acid formed is converted to yellow silicomolybdcic acid by the addition of ammonium molybdate to the solution. Spectrophotometric measurement of the yellow complex is then made at about 400 nm.

6.2 Reagents**6.2.1 Sodium Hydroxide Solution — 40 percent (m/v)**

Weigh 40 g of sodium hydroxide pellets in a plastic, polyethylene or stainless steel beaker and add 100 ml of water. Stir with a plastic rod until entire mass dissolves. Cool and store in a plastic or polythene bottle.

6.2.2 Dilute Nitric Acid — 1:1 (v/v).**6.2.3 Ammonium Molybdate Solution — 10 percent (m/v)**

Dissolve 100 g of ammonium molybdate in water and dilute to one litre. Filter before use.

6.2.4 Standard Silicon Solution (1 ml — 0.05 mg of Si)

Fuse 0.107 g of anhydrous silica with 1.0 g of sodium carbonate in a platinum crucible. Cool the melt, dissolve completely in water, and dilute to one litre in a volumetric flask. Store the solution in a polyethylene bottle.

NOTE — In case anhydrous silica is not available, adjust the weight of silica used according to the actual silicon content of the sample.

6.3 Procedure**6.3.1** Transfer 0.5 g of the accurately weighed sample to a 250 ml stainless steel or nickel beaker. Add 10 ml

of sodium hydroxide solution by means of a pipette. Cover the beaker and warm it to start the reaction. When the reaction subsides, wash the walls of the beaker and cover. Heat to complete the dissolution and boil for a few minutes. In case of alloys containing magnesium, concentrate the solution by boiling to ensure complete conversion of magnesium silicide to sodium silicate. Dilute the solution by washing the walls of the beaker and the cover with water. Cool and transfer the contents of the beaker to a Pyrex beaker containing 25 to 30 ml of dilute nitric acid (measured accurately). Transfer the contents of the metallic beaker completely to the glass beaker with a jet of water. Wash the metallic beaker three or four times with hot water, using a small quantity at a time, and transfer the washings to the glass beaker. Shake the contents of the beaker well and boil until the solution becomes clear (avoid excess boiling). Cool the solution.

6.3.2 Filter the solution through medium textured filter paper into a 250 ml volumetric flask. Wash the beaker 5 or 6 times with hot water. Dilute the solution to about 220 ml. Cool to room temperature, add 10 to 25 ml of ammonium molybdate solution and make up the volume to the mark. Shake the flask well. Allow the solution to stand for 15 min to develop the yellow colour fully.

6.3.3 Transfer a suitable portion of the solution to an absorption cell (4 cm) and adjust the spectrophotometer to the initial setting. Take the spectrophotometric readings within ten minutes of the development of colour, using a light filter at approximately 400 nm.

6.3.4 Calibration Curve

Take a series of standard silicon solutions and a reagent blank. Using same quantities of reagents, carry through all the stages as described under 6.3.2 and 6.3.3 and record the spectrophotometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the spectrophotometric readings of the standard solution against milligrams of silicon per 100 ml of the solution.

6.3.5 Convert the spectrophotometric readings of the sample to milligrams of silicon by means of calibration curve, and calculate the percentage of silicon as given in 6.4.

6.4 Calculation

$$\text{Silicon, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

- A* = mass in mg of silicon found in the aliquot solution, and
B = mass in g of the sample represented by the aliquot taken.

NOTE — In case of high manganese alloys, the solution after addition of nitric acid may be coloured brownish yellow. If so, bleach the colour by dropwise addition of saturated solution of sodium sulphite to the hot solution. Then add sufficient potassium permanganate solution (0.1 N) until a pink colour is obtained. Add finally just enough oxalic acid solution (one percent, *m/v*) to destroy the pink colour.

7 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD (FOR SILICON CONTENT OVER 0.3 PERCENT)

7.1 Outline of the Method

Silicon is converted to sodium silicate by alkaline attack. After acidification and removal of interfering elements, silica is determined by hydrofluorization of the residue.

7.2 Reagents

7.2.1 Sodium Hydroxide Solution — 50 percent (*m/v*).

7.2.2 Hydrogen Peroxide — 20 volumes.

7.2.3 Sulphuric Acid — r.d. = 1.84.

7.2.4 Sulphuric Acid — Perchloric Acid Mixture

Add 624 ml of sulphuric acid (r.d. = 1.84) and 85 ml of perchloric acid (70 percent) to 315 ml of water. Mix and make up to 1 litre.

7.2.5 Perchloric Acid Solution

Add 600 ml of perchloric acid (70 percent) to 400 ml of water and mix.

7.2.6 Nitric Acid — r.d. = 1.42.

7.2.7 Sulphurous Acid — Saturate water with sulphur dioxide gas. Prepare fresh as needed.

7.2.8 Sulphurous Acid — 1:1, 1:3, and 1:99 (*v/v*).

7.2.9 Hydrochloric Acid — r.d. 1.16.

7.2.10 Ammonium Acetate Solution

Dissolve 25 g of ammonium acetate in distilled water containing 1 ml of glacial acetic acid and make up to 100 ml with water.

7.2.11 Dilute Hydrochloric Acid — 1:99 (*v/v*).

7.2.12 Hydrofluoric Acid (40 percent)

NOTE — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membrane and produces severe skin burns which are slow to heal. In case of contact with skin, wash well with water and seek medical help immediately.

7.3 Procedure

7.3.1 Depending upon the silicon content of the sample, adjust the weight of the sample to be taken for analysis as given below. Accurately weigh the sample and transfer it to a stainless steel beaker or a nickel beaker:

Silicon Content Percent	Mass in g of the Sample to be Taken
Over 0.3 up to 1	2
Over 1 up to 3	1
Over 3 up to 7	0.5
Over 7	0.25

7.3.2 Add 12 ml of sodium hydroxide solution per gram of the sample taken. When the reaction is complete, wash down the cover and sides of the vessel with small jet of hot water. Keep the volume of the solution as low as possible. Place on the hot plate and evaporate cautiously to syrupy consistency. Add hydrogen peroxide dropwise (about 30 drops) and repeat the evaporation. Add more hydrogen peroxide dropwise until the solution is completely oxidized. Remove the vessel from hot plate, cool, wash the sides with 10 ml of sulphuric acid — perchloric acid mixture. Rinse the nickel vessel, first with hot water and then with a few millilitres of perchloric acid solution to which an equal volume of water has been added. Clean the vessel thoroughly by means of a rubber-tipped glass rod and finally rinse with hot water.

7.3.3 Add 5 ml of nitric acid and few ml of sulphurous acid to the solution, evaporate to dense white fumes, cover, fume for 15 min and cool. Add 100 ml of water and 10 ml of dilute sulphuric acid (1:3). Stir well and heat until the soluble salts are dissolved. If manganese, dioxide is present, bring it into solution with a few drops of hydrogen peroxide or sulphurous acid.

7.3.4 Add paper pulp, and filter on a low ash medium textures paper. Remove all silica from the beaker by means of rubber tipped glass rod and wash the paper and contents thoroughly with dilute sulphuric acid (1: 99). Recover silica from the filtrate by evaporating the filtrate, fuming and proceeding as under 7.3.3. Add the recovered silica to the main silica residue. Place the papers and precipitate in a platinum crucible, dry and ignite slowly at 500°C until free of carbon, then heat at 900 to 950°C for 25 min. Cool in a desiccator, weigh and again ignite at the same temperature as a check for constant mass.

7.3.5 If lead is present, wash the main silica residue (see 7.3.4) with hot ammonium acetate solution and preserve the ammonium acetate extract separately for determination of lead (see 8). Alternatively and

lead retained in the residue may be removed as under 7.3.5.1.

7.3.5.1 After fuming (see 7.3.3) take up the sample with 5 ml of hydrochloric acid and 100 ml of hot water. Filter and wash with hot dilute hydrochloric acid. The analyst may select the method for lead separation that best suits his plan for using the filtrate in subsequent analysis.

7.3.6 Moisten the residue with a few drops of water, then add 5 to 10 drops of dilute sulphuric acid (1:1) and a few millilitres of hydrofluoric acid. Evaporate to dryness, ignite at 900°C, cool and weigh. Repeat the treatment with hydrofluoric acid and ignite to constant weight. The loss in weight represents silica.

7.3.7 Make a blank determination, following the same procedure and using the same amounts of all reagents, but without the sample.

7.4 Calculation

$$\text{Total silicon, percent} = \frac{A - B}{C} \times 46.72$$

where

A = mass in g, of silica as obtained in 7.3.6 from the difference in weight before and after HF treatment;

B = mass in g, of silica obtained in the blank; and

C = mass in g, of the sample taken.

8 DETERMINATION OF LEAD BY THE SULPHIDE (COLORIMETRIC) METHOD

8.1 Outline of the Method

Lead is determined colorimetrically from the ammonium acetate extract obtained after silicon determination.

8.2 Reagents

8.2.1 *Dilute Nitric Acid* — 1:1 (v/v).

8.2.2 *Sucrose Solution* — 100 percent (m/v).

8.2.3 *Hydrogen Sulphide Solution*

Saturate dilute sulphuric acid (1:99) with hydrogen sulphide gas.

8.2.4 *Standard Lead Solution* (1 ml = 0.000 1 g of Pb)

Dissolve 0.1831 g of lead acetate crystals $[\text{Pb}(\text{CH}_3\text{CO}_2)_2 \cdot 3\text{H}_2\text{O}]$ in water containing a little acetic acid and dilute to one litre.

8.3 Procedure

Transfer the ammonium acetate extract preserved

under 7.3.5 to a 100 ml Nessler tube. Add water to make the solution to 90 ml. Add 3 drops of dilute nitric acid, one millilitre of sucrose solution and 5 ml of hydrogen sulphide solution. Make up the volume to 100 ml with water, and mix well. Compare the intensity of colour with those of series of standard solutions each containing same quantities of all the reagents as were added in the test solution. It is important to add hydrogen, sulphide solution, simultaneously to the standard and test solutions.

8.4 Calculation

$$\text{Lead, percent by mass} = \frac{M \times 0.1}{m}$$

where

M = mass in mg of lead found in the sample,
and

m = mass in g of the sample taken.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 2 DETERMINATION OF IRON

(Second Revision)

1 SCOPE

This standard (Part 2) describes the O-phenanthroline (Spectrophotometric) method of determination of iron up to 0.1 percent and (titrimetric) method above 0.1 percent in aluminium and its alloys.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below :

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

**5 DETERMINATION OF IRON BY
THE O-PHENANTHROLINE (SPECTRO-
PHOTOMETRIC) METHOD (FOR IRON
CONTENT BETWEEN 0.03 TO 0.10
PERCENT)**

5.1 Outline of the Method

All the trivalent iron in the sample solution is reduced by hydroxylammonium chloride to give bivalent iron, which is complexed with 1-10 phenanthroline. The complex gives an orange red colouration in acetate buffer at pH 3.5 to 4.2.

The intensity of the colour is measured at 510 nm spectrophotometrically.

5.2 Reagents**5.2.1 Hydroxylammonium Chloride Solution (10 percent)**

Dissolve 10 g hydroxylammonium chloride in 100 ml of distilled water. Store in a plastic container.

5.2.2 1-10 Phenanthroline Solution (0.25 percent)

Dissolve 0.25 g of 1-10 phenanthroline in minimum amount of ethyl alcohol and make the total volume to 100 ml with distilled water. Store in a plastic container.

5.2.3 Buffer Solution**5.2.3.1 Dissolve 272 g of sodium acetate in 500 ml of water. Add 240 ml of acetic acid. Make up the total volume to 1 000 ml by distilled water. Store in a plastic container.****5.2.3.2 Sodium hydroxide solution (15 percent)**

Dissolve 15g of sodium hydroxide pellets in distilled water. Cool and make up to 100 ml. Store in polyethylene container.

5.2.4 Aluminium Solution for Matrix Correction

Dissolve 1 000 g of aluminium metal (purity 99.99 percent) in 20 ml of sodium hydroxide (15 percent) and extract the content in 50 ml of 8N nitric acid. Make up the volume to 250 ml. Store in a plastic container.

5.2.5 Stock Iron Solution (1 000 mg/l)

Dissolve 1g of pure iron (99.99 percent) in 1:1 hydrochloric acid and make up to one litre.

5.2.5.1 Standard iron solution A (100 mg/l)

Using a pipette transfer 10 ml of the stock iron solution (1 000 ppm) to a 100 ml volumetric flask and make up the volume with distilled water.

5.2.5.2 Standard iron solution B (10 mg/l)

Pipette out 10 ml of standard iron solution (5.2.5.1) to a 100 ml volumetric flask and make up the volume to 100 ml with water.

5.3 Procedure

5.3.1 Follow the procedure specified in 5.2.1 to 5.3.3 of Part 1 for preparation of sample solution.

5.3.2 Add 5 ml of hydroxylammonium chloride solution and 5 ml of 1:10 phenanthroline solution and homogenize. Add 15 ml of the acetate buffer and make up the volume to 100 ml and homogenize. Measure the absorbance at 510 nm by a spectrophotometer using suitably prepared reagent blank.

5.3.3 Find the concentration of the sample solution from the calibration curve and calculate the percentage of iron as given in 5.3.5.

5.3.4 Calibration Curve

In a series of six volumetric flasks, add 1,2,3,4 and 5 ml of standard iron solution B to obtain 0.1, 0.2, 0.3, 0.4 and 0.5 mg/l of iron respectively. Use flask numbered '0' for reagent blank. Follow rest of the procedure as specified in 5.3.2.

5.3.5 Calculation

$$\text{Iron, percent by mass} = \frac{M \times 250}{m}$$

where

M = mass in mg/ml of sample found (in aliquot taken) from the calibration curve, and

m = mass in g, of the sample taken.

6 DETERMINATION OF IRON BY POTASSIUM DICHROMATE (TITRIMETRIC) METHOD (FOR IRON CONTENT > 0.10 PERCENT)

6.1 Outline of the Method

All the iron present in the sample is brought to solution by dissolving it in acid mixture. The trivalent iron if present, is reduced to bivalent by stannous chloride. The entire iron is oxidized by potassium dichromate.

6.2 Reagents

6.2.1 Acid Mixture

In a cleaned 2-litre glass beaker, take 1 000 ml distilled water. To it add 450 ml of sulphuric acid slowly with constant stirring and cooling under tap water. To the solution add 550 ml of hydrochloric acid slowly and stir to mix it completely.

6.2.2 Potassium Dichromate Solution (N/55.85)

Take 1.755 8 g of dried (at 110°C for 1 h) potassium dichromate and transfer it carefully to a 2-litre volumetric flask. Add 300 ml of water to it and shake until all the crystals dissolve. Then make up the volume with water and shake the entire solution for

complete mixing. It is desirable to add a small volume of H_2SO_4 while preparing the solution.

6.2.3 Stannous Chloride Solution (10 percent)

Dissolve 10 g of stannous chloride in 10 ml of hydrochloric acid by heating over a hot plate. Cool it and make up the volume to 100 ml with water. Store it in a plastic dropping bottle.

6.2.4 Mercuric Chloride Solution (Saturated)

Prepare a saturated solution of mercuric chloride with distilled water and store in a plastic container.

NOTE— Mercury and its compounds are highly poisonous. Take proper precautions while handling and disposing off them.

6.2.5 Sodium Diphenylamine Sulphonate Solution (0.20 percent)

Dissolve 0.2 g of sodium diphenylamine sulphonate in 100 ml of distilled water and store it in a dropping bottle.

6.2.6 Phosphoric-Sulphuric Acid Mixture

In a cleaned 1-litre beaker take 500 ml of distilled water. Add 250 ml of sulphuric acid slowly with constant stirring and cooling under tap water. To the cooled solution add 250 ml of phosphoric acid and homogenize.

6.3 Procedure

6.3.1 Take 1 000 g of sample drillings in a cleaned 500 ml conical flask. Add 50 ml of acid mixture and wait until the reaction is complete and the solution is clear. If some metal drillings remain unreacted, then heat gently over a hot plate until all the sample goes into the solution.

NOTE— If the colour of the solution is yellow then heat it over a hot plate and decolourize it with dropwise addition of stannous chloride solution. Adjust sufficient and do not add in excess.

6.3.2 Cool it immediately under tap water to room temperature. Add 10 ml of saturated solution of mercuric chloride at a time to it and shake gently. Silky white precipitate would appear. If it is black in colour, discard the solution and repeat the procedure from the beginning.

6.3.3 Add 10 ml of phosphoric-sulphuric acid mixture and 5-6 drops of sodium diphenylamine sulphonate indicator. Titrate against potassium dichromate solution to a suitable violet colouration.

7 STANDARDIZATION OF POTASSIUM DICHROMATE SOLUTION

Take 10 ml of standard iron solution 1 000 mg/l add 50 ml of acid mixture and heat it. If the colour is yellow, then decolourize as described above. Add

10 ml phosphoric-sulphuric acid mixture and 5-8 drops of sodium diphenylamine sulphonate indicator. Titrate against potassium dichromate solution to a stable violet colouration.

If the potassium dichromate solution is exactly N/55.85 then 10 ml of the 1 000 mg/l iron solution will consume 10 ml of the potassium dichromate solution.

If the potassium dichromate solution is not exactly N/55.85 then find out the factor (F) as follows:

$$F = \frac{10 \text{ ml}}{v}$$

where

v = the volume of dichromate consumed in ml.
1 000 ml of N/55.85 $K_2 Cr_2 O_7$ = 1g of iron

If the factor is 1.0 then

$$\text{Percent, iron in sample} = \frac{\text{ml of dichromate consumed}}{1 000 \times \text{mass of sample}} \times 100$$

otherwise

$$\text{percent, iron in sample} = \frac{\text{ml of dichromate consumed} \times F \times 100}{1 000 \times \text{mass of sample}}$$

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 3 DETERMINATION OF COPPER

(Second Revision)

1 SCOPE

This standard (Part 3) describes the methods for determination of copper up to 0.1 percent by the Diethyl-dithiocarbamate (spectrophotometric) method and above 0.1 percent by the iodimetric method in aluminium and its alloys.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
264 : 1976	Nitric acid (second revision)
265 : 1993	Hydrochloric acid (fourth revision)
266 : 1993	Sulphuric acid (third revision)
1070 : 1992	Reagent grade water (third revision)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (see IS 1070) shall be employed for the test.

5 DETERMINATION OF COPPER BY THE DIETHYL-DITHIOCARBAMATE (SPECTROPHOTOMETRIC) METHOD (FOR COPPER CONTENT UP TO 0.1 PERCENT)

5.1 Outline of the Method

The sample is treated with mixed acids and the silicon removed. Copper is precipitated as sulphide from the acid solution of the sample and any copper left in the residue is also recovered. The copper is dissolved in nitric acid, complexed with diethyl-dithiocarbamate and determined spectrophotometrically at approximately 435 nm.

5.2 Reagents

5.2.1 Mixed Acids

Add 170 ml of sulphuric acid (r.d. = 1.84) cautiously,

to 600 ml of water, cool and add 300 ml of hydrochloric acid (r.d. = 1.16) and 300 ml of nitric acid.

5.2.2 Dilute Sulphuric Acid — 1:1, 1:99 (v/v).

5.2.3 Hydrofluoric Acid (40 percent)

5.2.4 Hydrogen Sulphide — Gas.

5.2.5 Hydrogen Sulphide Wash Solution

Pass hydrogen sulphide gas through dilute sulphuric acid (1:99) to obtain a saturated solution for the existing.

5.2.6 Dilute Nitric Acid — 1:2 (v/v).

5.2.7 Ammonium Hydroxide (20 percent)

5.2.8 Dilute Ammonium Hydroxide Wash Solution — 1:20 (v/v).

5.2.9 Citric Acid Solution — 20 percent (m/v).

5.2.10 Sodium Diethyl-Dithiocarbamate Solution — 0.1 percent (m/v).

5.2.11 Carbon Tetrachloride

5.2.12 Sodium Sulphate — Solid.

5.2.13 Standard Copper Solution (1 ml = 0.01 mg of Cu).

Dissolve one gram of electrolytic copper in 10 ml of dilute nitric acid (1:1) evaporate nearly to dryness and dilute to 1 000 ml in a volumetric flask. Pipette 10 ml from this solution and dilute to one litre.

5.3 Procedure

5.3.1 Weigh accurately 2 g of sample and transfer to a 400 ml beaker. Add 10 ml of the mixed acids and after the reaction has subsided, add 60 ml more. Add 20 ml of dilute sulphuric acid (1:1) and evaporate the solution to fumes. Continue fuming for ten minutes. Cool, dilute to 200 ml and boil until soluble salts are dissolved, filter the insoluble matter on a paper pulp pad and wash with hot dilute sulphuric acid (1:99). Preserve the filtrate.

5.3.2 Ignite the residue in a silica basin at a temperature not exceeding 600°C. Transfer the residue to a platinum crucible. Moisten it with a little water. Add 2 to 3 drops

of dilute sulphuric acid (1:1) and 8 to 10 ml of hydrofluoric acid. Evaporate the dryness and ignite for about 5 min. dissolve in a few millilitres of water and mix with the filtrate preserved under 5.3.1.

5.3.3 Adjust the volume to 300 ml. Heat the solution to 60°C, add a small quantity of ashless paper pulp and pass hydrogen sulphide for about half an hour and keep for another half an hour at about 50 to 60°C. Filter through medium textured filter paper and wash 8 to 10 times with hydrogen sulphide wash solution. Transfer the filter paper along with the precipitate to the same beaker in which copper was precipitated. Add 30 to 40 ml of dilute nitric acid (1:2) and digest on a hot plate for 15 to 20 min. Dilute to 70-80 ml with hot water and make distinctly ammoniacal. Keep just boiling for 3 to 4 min. and filter through fine textured filter paper into a 200 ml volumetric flask. Wash with hot dilute ammonium hydroxide wash solution and discard the residue.

5.3.4 Pipette a suitable aliquot (depending on the copper content of the solution) into a 250 ml separating funnel. Add 10 ml of citric acid. Make distinctly ammoniacal. Add 10 ml of sodium diethyl-dithiocarbamate solution, mix well and allow to stand for 15 min. Extract successively with 25 ml, 15 ml and 10 ml of carbon tetrachloride. Combine carbon tetrachloride extracts and make up to 50 ml in a graduated flask. Add a little anhydrous sodium sulphate to clear the solution.

5.3.5 Transfer a suitable aliquot to an absorption cell and adjust the spectrophotometer to the initial setting. Take the spectrophotometric reading at 435 nm or using a photometer with a light filter at 435 nm.

5.3.6 Calibration Curve

Take a series of standard copper solutions and a reagent blank. Using same quantities of all reagents, carry through all the stages as described under 5.3.4 and 5.3.5 and record the spectrophotometric readings of the standard solutions along with the blank. Draw a calibration curve by plotting the spectrophotometric readings of standard solutions against milligrams of copper per 50 ml of organic extract of solution.

5.4 Calculation

Convert the spectrophotometric reading of the sample to milligrams of copper by means of calibration curve and calculate the percentage of copper as follows :

$$\text{Copper, percent by mass} = \frac{M}{m} \times \frac{1}{10}$$

where

M = mass in mg of copper found in the aliquot

solution, and

m = mass in g of the sample represented by the aliquot taken.

NOTE — Since the carbon tetrachloride extract of copper complex is readily bleached by exposure to strong light, extraction should be carried out in artificial light or weak daylight.

6 DETERMINATION OF COPPER BY THE IODIMETRIC METHOD (FOR COPPER CONTENT OVER 0.1 PERCENT)

6.1 Outline of the Method

The sample is treated with mixed acids, the silicon removed by fuming and subsequent filtration and copper in the filtrate is precipitated as sulphide. The copper sulphide is ignited and dissolved in acid and the copper determined iodimetrically. Antimony, if present, is separated from the copper sulphide in alkaline sulphide solution.

6.2 Reagents

6.2.1 Sulphuric Acid — r.d. = 1.84.

6.2.2 Nitric Acid — r.d. = 1.42.

6.2.3 Hydrochloric Acid — r.d. = 1.16.

6.2.4 Mixed Acids

To 700 ml of water add gradually 150 ml of sulphuric acid. Cool, then add 75 ml of nitric acid and 75 ml of hydrochloric acid.

6.2.5 Hydrofluoric Acid (40 percent.)

6.2.6 Dilute Sulphuric Acid — 1 : 1(v/v).

6.2.7 Ammonium Fluoride — Solid.

6.2.8 Ammonium Hydroxide (20 percent)

6.2.9 Acetic Acid — Glacial.

6.2.10 Urea Solution — 20 percent (m/v).

Dissolve 20 g of urea in 100 ml of distilled water.

6.2.11 Potassium Iodide Solution — 50 percent (m/v).

6.2.12 Standard Sodium Thiosulphate Solution (0.05 N and 0.02 N).

Dissolve 12.41 g of sodium thiosulphate in distilled water and make up to 1 000 ml. Stabilize by the addition of 2 to 3 grains of borax per litre. For preparing standard thiosulphate solution (0.02 N). Dilute 200 ml of standard thiosulphate solution (0.05) to 500 ml with water. This solution shall be used when the copper content is between 0.3 to 2.5 percent. Standardize the solution as described under 6.2.12.1.

6.2.12.1 Weigh out exactly one gram of electrolytic copper, dissolve in 10 ml of dilute nitric acid (1:1).

Boil nearly to dryness and dilute with water. Cool and make up the solution to 250 ml in a graduated flask. Pipette out exactly 25 ml (for standardizing 0.05 N solution). Add ammonium fluoride, ammonia, acetic acid, urea and potassium iodide as described under 6.3 and titrate with thiosulphate solution to be standardized, using starch as indicator.

6.2.13 Starch Solution

Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for 2 or 3 min and cool. Prepare the solution fresh as needed.

6.2.14 Ammonium Thiocyanate — Solid.

6.2.15 Hydrochloric Acid–Nitric Acid Mixture

Mix 500 ml of hydrochloric acid (6.2.3) with 500 ml of nitric acid (6.2.2).

6.2.16 Sodium Hydroxide Solution — 20 percent (m/v).

6.2.17 Sodium Sulphide Solution — 15 percent (m/v).

6.2.18 Sodium Hydroxide — Sodium Sulphide Wash Solution

Mix equal volumes of sodium hydroxide solution (one percent) and sodium sulphide solution (one percent).

6.2.19 Dilute Nitric Acid — 1 : 1 (v/v).

6.2.20 Sulphurous Acid

Saturate water with sulphur dioxide. Prepare fresh as needed.

6.2.21 Hydrogen Sulphide — Gas.

6.2.22 Hydrogen Sulphide Wash Solution

Saturate dilute sulphuric acid (1:99) with hydrogen sulphide gas.

6.3 Procedure (In the Absence of Antimony)

6.3.1 Take 2 g of the accurately weighed samples (see Note) in a 400 ml beaker and treat with 50 ml of mixed acids. After the reaction has subsided, add 20 ml more of the mixed acids, adding a little nitric acid if necessary. If silicon content is small (less than 2 percent), the recovery of copper from the residue is unnecessary provided that sufficient hydrofluoric acid is added at this stage to give a clear solution. Add 20 ml of dilute sulphuric acid, evaporate the solution to fuming and continue fuming for 10 min, cool, dilute to 200 ml and boil until the solution is clear. Filter off the insoluble matter on a paper pulp pad and wash with hot water. Precipitate copper by following the procedure given below:

Heat the filtrate to 60 to 70° and pass a steady stream

of hydrogen sulphide gas through the solution for 20 min. Add some ashless paper pulp and let stand for a few minutes to allow the precipitate to coagulate. Filter and wash the paper and precipitate 6 to 8 times with warm hydrogen sulphide wash solution.

6.3.2 Dry and ignite the copper sulphide residue at not more than 700°C in a porcelain or silica crucible. Moisten the residue in the crucible with a few drops of water and warm with 2 to 3 ml of nitric acid to re-dissolve. Transfer to 300 ml of conical flask or beaker, boil nearly to dryness to remove most of the excess nitric acid and re-dissolve in approximately 10 ml of water.

6.3.3 Add 0.1 to 0.2 g of ammonium fluoride, neutralize carefully by adding ammonium hydroxide drop by drop until blue complex is formed, and then add one drop in excess. Acidify with glacial acetic acid added dropwise, and add ten drops in excess, followed by a drop of urea solution. Wash the sides of the flask or beaker using not more than 5 ml of water. Add 5 ml of potassium iodide solution and titrate with standard sodium thiosulphate solution adding one millilitre starch solution as indicator towards the end of the titration. Add about one to two grams of ammonium thiocyanate (to liberate the absorbed iodine) and continue the titration until the blue colour is completely discharged.

6.3.4 Calculation

$$\text{Copper, percent by mass} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml, of the standard sodium thiosulphate solution required to titrate the sample;

B = copper equivalent of the standard sodium thiosulphate solution in g/ml; and

C = mass in g, of the sample taken.

6.4 Procedure (In the Presence of Antimony and Tin)

6.4.1 Treat 2 g of the accurately weighed sample in a 600 ml beaker with 40 ml of hydrochloric acid–nitric acid mixture. When the reaction is complete, dilute slightly and evaporate by boiling until just pasty. Dissolve in a small amount of water and pour into 100 ml of sodium hydroxide solution with constant stirring. Dilute to 300 ml, bring to the boil, and 25 ml of sodium sulphide wash solution, re-boil and allow the precipitate to settle for 30 min. Filter on a paper pulp pad and wash with sodium hydroxide-sodium sulphide wash solution.

6.4.2 Extract the residue with hot dilute nitric acid adding a little sulphurous acid, if necessary, and wash with hot water. Add 5 ml of sulphuric acid and evaporate by boiling to a low bulk. Dry the paper pulp pad, from which the bulk of the copper sulphide has been extracted and ignite for 20 to 30 min in a platinum crucible at approximately 700°C. Dissolve the residue in a few drops of nitric acid, add a few drops of hydrofluoric acid, if necessary, to remove the silicon, combine with the sulphuric acid solution obtained above and evaporate the solution to fuming, cool, dilute

with 250 ml of hot water, heat to 60°C and saturate with hydrogen sulphide gas. Filter off the copper sulphide and wash with hydrogen sulphide wash solution, dry and ignite the paper and the residue as before. Moisten the residue in the crucible with a few drops of water and warm with 2 to 3 ml of nitric acid to dissolve. Transfer to a conical flask or beaker, boil nearly to dryness and dissolve in a small amount of water. Proceed further as described under 6.3.3 and 6.3.4.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 4 DETERMINATION OF ZINC

(Second Revision)

1 SCOPE

This standard (Part 4) describes the method for determination of zinc up to 0.1 percent by the Dithizone (Spectrophotometric) method and by Mercuric Thiocyanate (Volumetric) method and above 0.1 percent by Mercuric Thiocyanate (Gravimetric) method in aluminium and its alloys.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see IS 1070*) shall be employed for the test.

5 DETERMINATION OF ZINC BY THE DITHIZONE (PHOTOMETRIC) METHOD (FOR ZINC CONTENT BELOW 0.1 PERCENT)

5.1 Outline of the Method

The sample is dissolved in hydrochloric acid and potassium chlorate. Red colour complex obtained by the addition of dithizone to an aliquot of the solution is extracted with carbon tetrachloride and determined spectrophotometrically at about 530 nm.

5.2 Reagents

5.2.1 Dilute Hydrochloric Acid — 1 : 1 (v/v).

5.2.2 Potassium Chlorate — Solid.

5.2.3 Carbon Tetrachloride

5.2.4 Complex – Forming Solution

Add successively, with shaking, to a 500 ml separator funnel, 15 drops of ammonium hydroxide, 1.5 ml of ammonium oxalate solution (5 percent), 15 ml of sodium acetate (5 percent), 35 ml of hydrochloric acid (1N), and a mixture of 120 ml of sodium thiosulphate solution (50 percent) and 150 ml of sodium sulphide solution (one percent). Shake the solution with small portions of dithizone until the latter remains green. Since the extraction of zinc proceeds slowly, it is better to use a shaking machine and allow 10 min for the reaction. Remove droplets of dithizone by finally washing with carbon tetrachloride. Slight separation of sulphur in aqueous solution does no harm.

5.2.5 Dithizone Solution

Dissolve 0.01 g of reagent in 100 ml of carbon tetrachloride.

5.2.6 Sodium Sulphide Wash Solution

Dissolve 5 g of sodium sulphide in 500 ml of water. Dilute 25 ml of the solution to 500 ml.

5.2.7 Standard Zinc Solution (1 ml = 0.01 mg of Zn)

Dissolve 0.1 g of pure zinc (purity 99.95 percent) in 5 to 10 ml of dilute hydrochloric acid (1:1). Cool and dilute to one litre in a volumetric flask. Transfer 10 ml of this solution to a 100 ml volumetric flask and make up the volume to the mark.

5.2.8 Ammonium Hydroxide (20 percent)

5.2.9 Ammonium Oxalate Solution (5 percent)

5.2.10 Sodium Acetate Solution (5 percent)

5.2.11 Sodium Thiosulphate Solution (50 percent)

5.3 Procedure

5.3.1 Dissolve 0.25 g of the accurately weighed sample in 12.5 ml of dilute hydrochloric acid, add 0.2 g of

potassium chlorate and boil for 5 min. Dilute the solution to 250 ml in the volumetric flask and filter through coarse paper, rejecting the first portion of the filtrate.

5.3.2 Pipette a suitable aliquot (depending upon the zinc content of the sample) into a 100 ml separator funnel, add 30 ml of complex forming solution and 10 ml of dithizone solution and shake vigorously. If the carbon tetrachloride becomes red or red-violet in a short time, draw off the extract into a graduated glass stoppered, 25 ml cylinder and shake the aqueous phase with a fresh 5 ml portion of dithizone solution. If after 5 min the carbon tetrachloride is only a dark violet, separate it, add 1 ml of dithizone solution and shake for a minute. The last extract shall remain green. Wash the last extract with carbon tetrachloride.

5.3.3 Add 5 ml of the sodium sulphide wash solution to the combined extract and dilute the latter to 25 ml with carbon tetrachloride. Shake a few times with the wash solution and draw off the carbon tetrachloride phase. Repeat the washings three to four times with 5 ml portions of the wash solution. The last portion of the wash solution shall remain colourless. Filter the carbon tetrachloride extract through an acid-free, filter paper of coarse texture to remove water droplets. Transfer a suitable portion of the solution to an absorption cell and take the photometric readings using a light filter at 530 nm or use a spectrophotometer to measure the absorbance at 530 nm.

5.3.4 Calibration Curve

Take a series of standard zinc solutions and the reagent blank. Using the same quantities of reagents, carry through all stages as described under **5.3.2** and **5.3.3** and record the spectrophotometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the spectrophotometric readings of standard solutions against milligrams of zinc per hundred millilitres of the solution.

5.4 Calculation

Convert the spectrophotometric reading of the sample to milligrams of zinc by means of calibration curve, and calculate the percentage of zinc as follows:

$$\text{Zinc, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg, of zinc found in the aliquot of the sample solution used; and

B = mass in g of the sample represented by the aliquot taken.

6 DETERMINATION OF ZINC BY MERCURIC THIOCYANATE METHOD

6.1 Outline of the Method

The sample is dissolved in mixed acids, silicon and copper precipitated by fuming and by means of hydrogen sulphide respectively. From the filtrate zinc is precipitated as sulphide in the presence of formic acid. Zinc sulphide is extracted with acid and zinc mercuric thiocyanate precipitated from the extract, dried and weighed or titrated with standard potassium iodate solution depending upon the zinc content.

6.2 Reagents

6.2.1 Mixed Acid

Add 150 ml of sulphuric acid (r.d. = 1.84) cautiously to 700 ml of water and cool. Then add 75 ml of hydrochloric acid (r.d. = 1.16) and 75 ml of nitric acid (r.d. = 1.42).

6.2.2 Dilute Sulphuric Acid — 1 : 1 (v/v).

6.2.3 Hydrogen Sulphide — Gas.

6.2.4 Hydrogen Sulphide Wash Solution

Saturate sulphuric acid (one percent) with hydrogen sulphide gas.

6.2.5 Tartaric Acid Solution — 30 percent (v/v).

6.2.6 Ammonium Hydroxide (20 percent)

6.2.7 Methyl Red Indicator Solution — 0.1 percent (m/v)

Dissolve 0.1 g of methyl red in 3.72 ml of sodium hydroxide solution (0.1 N) and dilute to 250 ml with water. Filter, if necessary.

6.2.8 Formic Acid Mixture

To 20 ml of formic acid (r.d. = 1.20), add 25 g of ammonium sulphate and 3 ml of ammonium hydroxide and dilute to 100 ml.

6.2.9 Formic Acid Wash Solution

Dilute 25 ml of formic acid mixture to one litre and saturate with hydrogen sulphide gas.

6.2.10 Dilute Hydrochloric Acid — 1:1 (v/v).

6.2.11 Nitric Acid — r.d. = 1.42.

6.2.12 Ammonium Nitrate — Solid.

6.2.13 Methylated Spirit

6.2.14 Sulphuric Acid — Methylated Spirit Mixture

Add one millilitre of sulphuric acid (r.d. = 1.84) to 200 ml of water and then add 200 ml of industrial methylated spirit.

6.2.15 Mercuric Potassium Thiocyanate Solution

Dissolve 54 g of mercuric chloride in water, add to 76 g of potassium thiocyanate dissolved in water and dilute to two litres. Filter before use.

6.2.16 Concentrated Hydrochloric Acid

6.2.17 Chloroform

6.2.18 Standard Zinc Solution (1 ml = 0.000 1 g of Zn)

Dissolve 0.1 g pure electrolytic zinc in sufficient dilute sulphuric acid (1:1) and dilute to 1 000 ml with water.

6.2.19 Standard Potassium Iodate Solution

Dissolve 9.820 g of potassium iodate in water and dilute to 1 000 ml. Pipette three portions each of 20 ml of standard zinc solution into separate 200 ml beakers. To each add 5 ml of dilute sulphuric acid (1:1), dilute to 75 ml with water and precipitate with 25 ml of mercuric potassium thiocyanate solution. Filter, wash the precipitates. Extract and titrate each extract separately with standard potassium iodate solution as described in 6.3. From the mean titre adjust the strength of the potassium iodate solution so that 1 ml = 0.000 1 g, 0.000 2 g or 0.000 5 g of zinc depending on the amount of zinc expected in the sample.

6.3 Procedure (Gravimetric Method) (for Zinc Content Above 0.1 Percent)

6.3.1 Depending upon the zinc content of the sample, adjust the mass of the sample and volume of the mixed acid as given below to a conical weighed beaker:

Zinc Content, percent	Mass in g of the Sample to be Taken	Volume in ml of Mixed Acid Required
Over 2 up to 4	0.5	25
Over 1 up to 2	1	35
Over 0.1 up to 1	2	70
Over 0.05 up to 0.1	3	90
Up to 0.05	5	100

6.3.2 Cool, if necessary. Add 20 ml of dilute sulphuric acid carefully, evaporate to fumes and continue fuming for 10 min. cool, dilute to 200 ml with water and boil until solution is complete. Allow it to cool. Cool to 60 to 70°C and pass hydrogen sulphide rapidly through the solution for approximately 20 min. Allow the precipitate to coagulate. Immediately filter the silica and copper sulphides on a paper pulp pad, wash with warm hydrogen sulphide wash solution.

6.3.3 Boil the filtrate to remove all hydrogen sulphide gas and adjust the volume to about 250 ml. Add 25 ml of tartaric acid solution and neutralize with ammonium hydroxide using methyl red as indicator. Add 25 ml

of formic acid mixture, heat to 70 to 80°C. Add a little paper pulp and pass hydrogen sulphide gas rapidly through the solution for 30 min. Allow the precipitate of zinc sulphide to coagulate, filter on a pulp pad and wash with warm formic acid wash solution. Extract the precipitate with dilute hydrochloric acid and wash with hot water. Add 5 ml of nitric acid and 10 ml of dilute sulphuric acid. Evaporate to fuming, decolourize with a few crystals of ammonium nitrate and fume for 10 min. Wash down and re-fume for 10 min, cool, dilute to 50 ml with water, add 50 ml of methylated spirit, mix well and allow to stand overnight. Filter the lead sulphate through a sintered glass filter funnel containing paper pulp pad using gentle suction, and wash with cold sulphuric acid-methylated spirit mixture.

6.3.4 Evaporate the filtrate to dryness, add 35 ml of water and 10 ml of dilute sulphuric acid, boil until clear. Cool and precipitate the zinc with 25 ml of mercuric potassium thiocyanate solution with vigorous stirring. Allow to stand for at least 8 h and filter by suction on a weighed sintered-glass crucible. Wash 6 times (25 ml each time) with mercuric potassium thiocyanate solution, diluted 50 times. Dry at 110°C to constant weight.

6.3.5 Carry out a blank determination, following the same procedure and using the same amounts of all reagents, but without the sample.

6.3.6 Calculation

$$\text{Zinc, percent by mass} = \frac{(A - B)}{C} \times 13.12$$

where

- A* = mass in g, of zinc mercuric thiocyanate,
B = correction for the blank in g, and
C = mass in g, of the sample taken.

6.4 Procedure [By Mercuric Thiocyanate (Volumetric) Method (for Zinc Content up to 0.1 Percent)]

6.4.1 Take the filtered and washed zinc mercuric thiocyanate precipitate as obtained under 6.3.4 in a 60 ml of hydrochloric acid added round the edge of the sintered-glass filter funnel. Allow to stand for 1 min and then apply suction. Add further 2 ml of hydrochloric acid dropwise as before, wash alternately with 1 ml of water and hydrochloric acid. Finally pour 0.5 ml of water. Add 0.5 to 1 ml of chloroform to the solution in the bottle and titrate with standard potassium iodate solution until the chloroform layer is just colourless. In most cases the titration is started by adding 4 drops of iodate solution and then shaking. Further additions are then made drop by drop with thorough shaking.

6.4.2 Calculation

$$\text{Zinc, percent by mass} = \frac{(A - B)C}{D} \times 100$$

where

A = volume in ml, of the standard potassium iodate solution used in the test;

B = volume in ml, of the standard potassium iodate solution required for the blank;

C = zinc equivalent of the standard potassium iodate solution in g/ml; and

D = mass in g, of the sample taken.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 5 DETERMINATION OF MANGANESE

(Second Revision)

1 SCOPE

This standard (Part 5) describes method for determination of manganese content in the range 0.1 to 1.5 percent by the Bismuthate method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
264 : 1976	Nitric Acid (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see IS 1070*) shall be employed for the test.

5 DETERMINATION OF MANGANESE BY THE BISMUTHATE METHOD (FOR MANGANESE CONTENT BETWEEN 0.01 TO 1.5 PERCENT)

5.1 Outline of the Method

Manganese in the solution of the sample is oxidized to permanganic acid by means of sodium bismuthate in nitric acid medium and the permanganate thus formed is titrated with standard ferrous ammonium sulphate solution.

5.2 Reagent

5.2.1 Nitric acid — r.d. = 1.42.

5.2.2 Sodium Bismuthate — Solid.

5.2.3 Sulphurous Acid

Make approximately 5 percent solution of sulphur dioxide in water.

5.2.4 Dilute Nitric Acid — 3:97 (v/v).

5.2.5 Phosphoric Acid — 85 percent (m/v).

5.2.6 Standard Ferrous Ammonium Sulphate Solution (0.03 N)

Dissolve 6 g of ferrous ammonium sulphate $[Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O]$ in cold dilute sulphuric acid (5:95) and make the volume to 500 ml with this acid. Mix well and keep in an amber-coloured glass-stoppered bottle.

5.2.7 Standard Sodium Oxalate Solution

Dissolve 2.01 g of sodium oxalate in water and make up to one litre in a graduated flask.

5.2.8 Standard Potassium Permanganate Solution (0.030 N)

Dissolve 0.958 g of potassium permanganate in water and dilute to 1 000 ml. Set the solution aside for two weeks in a dark place, filter without washing through a fritted glass crucible and store in a dark-coloured stoppered bottle. Standardize the permanganate solution against standard sodium oxalate solution (*see 7.2.7*).

5.3 Procedure

5.3.1 Depending upon the manganese content of the sample, transfer an accurately weighed sample and the volume of sodium hydroxide, in solution as given below to a 500 ml beaker :

Percent by Mass	the Sample to be Taken	Volume in ml of Sodium Hydroxide Solution Required
-----------------	------------------------	--

Over 1.0 up to 1.5 1 25

Over 0.25 up to 1.0 3 50

Over 0.1 up to 0.25 5 100

Treat the sample with 25 ml of sodium hydroxide solution (50 ml for 2 g of sample) and bring to boiling point on a hot plate until all visible action ceases.

(For silicon content greater than 4 percent, concentrate the solution at this stage to about half volume). Cool slightly, then add 60 ml of the acid mixture (100 ml for a 2 g sample) wash down the sides of the beaker and boil gently until solution is complete and nitrous fumes are expelled.

5.3.2 Dissolve the sample as described under **5.3.1**. Neutralize the solution with nitric acid and add 40 ml in excess. Boil the solution and add 0.5 g of sodium bismuthate. Boil for 2 to 3 min and dissolve the precipitated oxides of manganese in sufficient amount of sulphurous acid, added dropwise till solution is clear. Boil to expel oxides of sulphur and nitrogen. Dilute to about 100 ml with water and cool the solution to 10 to 15°C.

5.3.3 Add about 0.5 g of sodium bismuthate (or 26 times more than manganese content of the sample but not less than 0.5 g). Agitate for 2 or 3 min and filter immediately through asbestos Gooch crucible. Wash with cold dilute nitric acid until the washings are colourless. Reject the insoluble residue and maintain the temperature of the filtrate at 10 to 15°C. Add 2 ml of syrupy phosphoric acid to the measured excess of standard ferrous ammonium sulphate

solution. Titrate immediately the excess of ferrous iron with the standard permanganate solution to the faint pink colour.

5.3.4 Carry out a blank determination following the same procedure and using the same amount of the standard ferrous ammonium sulphate solution and other reagent as were used in the titration, but without the sample.

5.4 Calculation

$$\text{Manganese, percent} = \frac{(A - B) C \times 1.1}{D}$$

where

A = volume, in ml of the standard permanganate solution required to titrate the blank;

B = volume, in ml of the standard permanganate solution required to titrate the excess of ferrous ammonium sulphate solution;

C = normality of the standard permanganate solution; and

D = mass, in g of the sample taken.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 6 DETERMINATION OF MAGNESIUM

(Second Revision)

1 SCOPE

This standard (Part 6) describes the method for determination of magnesium in the range from 0.01 to 12 percent by the oxine method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below :

IS No.	Title
265 : 1987	Hydrochloric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see IS 1070*) shall be employed for the test.

5 DETERMINATION OF MAGNESIUM BY THE OXINE METHOD (FOR MAGNESIUM CONTENT BETWEEN 0.01 TO 12 PERCENT)

5.1 Outline of the Method

After the removal of interfering elements, magnesium in the solution of the sample is precipitated with 8 hydroxyquinoline and determined either gravimetrically or volumetrically.

5.2 Reagents

5.2.1 Sodium Hydroxide — Solid.

5.2.2 Hydrogen Peroxide — 20 volumes.

5.2.3 Sodium Carbonate — Solid.

5.2.4 Sodium Carbonate Solution — 5 g/l.

5.2.5 Methyl Red Indicator Solution — 0.1 percent (*m/v*)

Dissolve 0.1 g of methyl red in 3.72 ml of sodium hydroxide solution (0.1 N) and dilute to 250 ml with water. Filter, if necessary.

5.2.6 Ammonium Hydroxide (20 percent)

5.2.7 Ammonium Chloride Wash Solution

Dissolve 10 g of ammonium chloride in water, dilute to one litre, and make just yellow to methyl red with ammonium hydroxide.

5.2.8 Ammonium Chloride — Solid.

5.2.9 Hydrochloric Acid — r.d. = 1.16.

5.2.10 Ammonium Sulphide Solution

Saturate ammonium hydroxide (1:9) with hydrogen sulphide gas.

5.2.11 Ammonium Persulphate — Solid.

5.2.12 Ammonium Sulphide Wash Solution

Add 10 ml of ammonium sulphide solution to one litre of water containing 10 g of ammonium chloride and a few drops of ammonium hydroxide.

5.2.13 Bromine Water — Saturated.

5.2.14 8 Hydroxyquinoline Solution

Dissolve 5 g of 8 hydroxyquinoline in 10 ml of glacial acetic acid and dilute to 100 ml with water.

5.2.15 Dilute Ammonium Hydroxide — 1:99 (*v/v*).

5.2.16 Methyl Orange Indicator Solution

Dissolve 0.05 g of methyl orange in 100 ml of alcohol.

5.2.17 Potassium Bromate — **Potassium Bromide Solution**

Dissolve 2 g of potassium hydroxide, 80 g of potassium bromide, and 9 g of potassium bromate, in 700 ml of water and dilute to one litre.

5.2.18 Potassium Iodide Solution — 300 g/l (*m/v*).

5.2.19 Starch Solution

Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for 2 to 3 min and cool. Prepare the solution fresh as needed.

5.2.20 Standard Potassium Iodate Solution (0.33 N)

Dissolve 11.771 g of the potassium iodate dried at 180°C to constant mass in 200 ml of water, containing 1 g of sodium hydroxide and add 10 g of potassium iodide; when solution is complete, dilute to one litre in a volumetric flask.

5.2.21 Standard Sodium Thiosulphate Solution (0.33 N)

Dissolve 81.84 g of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in one litre of freshly boiled and cooled water in a sterile glass bottle. Standardize as follows:

Pipette 25 ml of the standard potassium iodate solution (see 5.2.20) into a 125 ml flask. Add 30 ml of water, one gram of the potassium iodide and 10 ml of dilute sulphuric acid (1 : 4). Titrate with sodium thiosulphate solution to a light straw colour. Add 2 ml of starch solution and continue titration to the disappearance of the blue colour.

5.3 Procedure

5.3.1 Depending upon the magnesium content of the sample, transfer an accurately weighed sample as given below to a 250 ml wide mouthed Erlenmeyer flask:

Magnesium Content, Percent	Mass in g of the Sample to be Taken
Over 2.0	0.5 or less
Over 0.2 up to 2.0	1
Over 0.01 up to 0.2	2

It is recommended that for high magnesium content, such a mass of sample be taken that it does not contain more than 0.05 g of magnesium.

5.3.2 Dissolve the sample in sodium hydroxide solution (see Note 1), using about 6 g of sodium hydroxide per gram of the sample. After violent reaction has subsided, heat the flask to hasten the decomposition of the sample. When visible evolution of hydrogen ceases, cautiously add few drops of hydrogen peroxide to the hot concentrated solution to complete the oxidation of silicon, zinc and chromium (see Note 2). Dilute to 150 ml and add 3 to 5 g of sodium carbonate. Stir well, and allow the precipitate to settle. Filter and wash the residue with hot sodium carbonate solution. Dissolve the magnesium in the residue, into original container, with 40 ml of dilute

hydrochloric acid (1:1) and wash the filter with hot water.

NOTES

1 In case of high silicon alloys, sample should be first attacked with weak alkali and subsequently by strong alkali. By the former treatment aluminium will dissolve almost completely and with the latter treatment silicon will dissolve without precipitation of silicates.

2 If chromium-bearing constituents are not completely decomposed, an insoluble residue containing some magnesium will remain after treatment with concentrated hydrochloric acid.

5.3.3 If the sample does not contain manganese, nickel or more than 3 mg of copper, add few drops of methyl red indicator and then add ammonium hydroxide and about 5 g of ammonium chloride until the solution is yellow to methyl red. Allow the precipitate to coagulate, filter and wash with hot ammonium chloride wash solution. If the magnesium content is over 0.02 g, place the paper with the precipitate in the original container and dissolve with 15 ml of dilute hydrochloric acid. Dilute to 50 ml with hot water. Precipitate as before, filter and wash with hot ammonium chloride wash solution. Make the combined filtrate just acid to methyl red and precipitate magnesium as before.

5.3.4 In case the sample contains manganese, nickel and more than 3 mg of copper, neutralize the acid solution as obtained under 5.3.2 with ammonium hydroxide and add about 5 g of ammonium chloride. Add 5 ml of ammonium sulphide solution (see Note). Filter into a 250-ml beaker, and wash with ammonium sulphide wash solution. If more than 20 mg of magnesium are present in the sample, some magnesium may be carried down with the sulphides. In that case, dissolve the precipitate with 15 ml of dilute hydrochloric acid and re-precipitate as under 5.3.3. Filter and wash the sulphides. Combine the filtrates and boil vigorously to drive off the ammonium sulphide. If a noticeable sulphide precipitate forms as the solution boil and becomes acid (usually indicating nickel or tin), filter, wash and boil the filtrate. Oxidize the remaining sulphur by adding an excess of bromine water, and continue boiling until the colour disappears.

NOTE — In case of alloys with manganese content over one percent, manganese may not completely precipitate by ammonium sulphide. In that case, add a few grams of ammonium persulphate to the filtrate to separate manganese completely.

5.3.5 Adjust the volume of the solution obtained under 5.3.3 or 5.3.4 to 75 ml. Remove from the hot plate, add 10 ml of bromine water, and while stirring make the solution alkaline with ammonium hydroxide and add 15 drops in excess. Digest until any manganese present precipitates as manganese dioxide, keeping the solution ammoniacal. Stir just as the solution begins to boil to aid coagulation. Filter and wash with hot water.

5.3.6 Make the filtrate acidic to methyl red with hydrochloric acid. If the indicator does not hold its colour, boil the solution until the bromine is gone. Make the volume of the solution to about 150 ml, neutralize the solution with ammonium hydroxide. Add 5 ml of ammonium acetate and 4 ml of acetic acid. Warm to 70°C, treat with slight excess of oxine solution. Filter and to the filtrate add ammonium hydroxide in excess. Add 5 ml of oxine solution, while stirring (5 ml is enough for 0.015 g of magnesium; for larger amounts of magnesium up to 0.03 g, add 10 ml). Heat the solution just to boiling, remove from the source of heat and allow to stand for 15 to 20 min. Filter and wash with hot dilute ammonium hydroxide. Since only magnesium oxinate is present, the amount may be determined either gravimetrically or volumetrically as described under **5.3.7** and **5.3.8** respectively.

5.3.7 Gravimetric Method

Filter the magnesium oxinate precipitate on a weighed sintered-glass funnel, washing with hot weakly ammoniacal water, dry at 100 to 105°C and weigh as the dihydrate. If the quantity of the oxinate is small, proceed as under **5.3.8**.

5.3.7.1 Calculation

$$\text{Magnesium, percent by mass} = \frac{A}{B} \times 6.98$$

where

A = mass in g of the oxinate, and

B = mass in g of the sample taken.

5.3.8 Volumetric Method

Dissolve the precipitate of oxinate as obtained under **5.3.7** into a 400 ml stoppered bottle using 30 ml of

hot dilute hydrochloric acid. Wash with cold dilute hydrochloric acid (90 ml of water plus 35 ml of dilute hydrochloric acid) and then with hot water so as to give an acid ratio of 1:4 at a volume of approximately 150 ml. Add a few drops of methyl orange indicator solution. Run in potassium bromate-potassium bromide solution from a burette, until the indicator is decolorized (indicating that bromate is present in excess) and add 5 ml more. Note the volume of the potassium bromate-potassium bromide solution added. Then add 10 ml of potassium iodide solution. Titrate immediately with the standard sodium thiosulphate solution using 2 ml of starch solution as an indicator near the end of the titration. The time from the addition of potassium bromate-potassium bromide solution to the end of the titration should not exceed 2 min.

5.3.8.1 Carry out a blank using the same quantity of the reagents. Titrate the blank through each step of procedure.

5.3.8.2 Calculation

$$\text{Magnesium, percent by mass} = \frac{(A - B) C \times 0.3}{D}$$

where

A = volume in ml of the standard sodium thiosulphate solution required to titrate the blank;

B = volume in ml of the standard sodium thiosulphate solution required to titrate the sample;

C = normality of the standard sodium thiosulphate solution; and

D = mass, in g of the sample taken.

*Indian Standard***CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS****PART 7 DETERMINATION OF NICKEL***(Second Revision)***1 SCOPE**

This standard (Part 7) describes the method for determination of nickel up to 4 percent by the dimethylglyoxime (gravimetric) method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
264 : 1976	Nitric Acid (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

**5 DETERMINATION OF NICKEL BY THE
DIMETHYLGlyOXIME (GRAVIMETRIC)
METHOD****5.1 Outline of the Method**

After removal of the interfering elements, nickel present in the sample solution is precipitated in ammoniacal medium by dimethylglyoxime, filtered, washed, dried and weighed.

5.2 Reagents

5.2.1 Sodium Hydroxide Solution — 20 percent (v/v).

5.2.2 Dilute Hydrochloric Acid — 1:1 (v/v).

5.2.3 Nitric Acid — r.d. = 1.42.

5.2.4 Dilute Sulphuric Acid — 1:1 (v/v).

5.2.5 Hydrogen Sulphide — Gas.

5.2.6 Hydrogen Sulphide Wash Solution

Saturate dilute sulphuric acid (1:99) with hydrogen sulphide.

5.2.7 Tartaric Acid Solution

Dissolve 25 g of tartaric acid in 60 ml of water, filter, add 1 ml of nitric acid and dilute to 100 ml.

5.2.8 Ammonium Chloride — Solid.

5.2.9 Ammonium Hydroxide (20 percent)

5.2.10 Dimethylglyoxime Solution

Dissolve 1 g of dimethylglyoxime in 100 ml of ammonium hydroxide and filter.

5.3 Procedure

5.3.1 Depending upon the nickel content of the sample, transfer an accurately weighed sample and the volume of alkali to the beaker as per the schedule give below:

Nickel Content, Percent	Mass in g of the Sample to be Taken	Volume in ml of Sodium Hydroxide Solution Required
From 0.5 up to 4	2	20
From 0.1 up to 0.5	5	40
Less than 0.1	10	100

5.3.2 Dissolve the sample in the required amount of sodium hydroxide solution. When the reaction is complete, dilute to 150 ml with hot water, allow the residue to settle for a short time, filter and wash with hot water. Dissolve the residue with a minimum amount of hot hydrochloric acid together with a little nitric acid, wash the paper thoroughly with hot water. Add 10 ml of dilute sulphuric acid, evaporate to dense white fumes, dilute to 100 ml with hot water, and saturate with hydrogen sulphide gas, filter and wash with hydrogen sulphide wash solution.

5.3.3 Boil the filtrate to remove the hydrogen sulphide

gas. Add 5 ml of nitric acid, and boil to oxidize iron. Dilute to 200 ml, add 10 ml of tartaric acid solution and 5 g of ammonium chloride. Neutralize with ammonium hydroxide. Acidify with dilute hydrochloric acid (1:1). Heat nearly to boiling and while stirring add dimethylglyoxime solution (20 ml for nickel up to 0.02 g and 5 ml for each additional 0.01 g). Allow to stand for 1 h. Filter and wash with cold water. Dry the precipitate at 110 to 120°C to constant mass. Cool in a desiccator and weigh as nickel

dimethylglyoxime.

5.4 Calculation

$$\text{Nickel, percent by mass} = \frac{A \times 20.32}{B}$$

where

A = mass in g, of nickel dimethylglyoxime, and
 B = mass in g, of the sample taken.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 8 DETERMINATION OF CHROMIUM

(Second Revision)

1 SCOPE

This standard (Part 8) describes the method for determination of chromium in aluminium and its alloys.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
264 : 1976	Nitric acid (<i>second revision</i>)
266 : 1993	Sulphuric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* 1070) shall be employed for the test.

5 DETERMINATION OF CHROMIUM BY THE PERSULPHATE OXIDATION (VOLUMETRIC) METHOD

5.1 Outline of the Method

Chromium is oxidized to chromate by means of ammonium persulphate in presence of silver nitrate and the potassium permanganate formed is destroyed with hydrochloric acid. It is then reduced with a measured volume of ferrous ammonium sulphate solution and the excess ferrous solution is titrated with standard permanganate solution.

5.2 Reagents

5.2.1 Sulphuric Acid — r.d. = 1.84.

5.2.2 Nitric Acid — r.d. = 1.42.

5.2.3 Sulphuric Acid — Nitric Acid Mixture

Slowly stir 100 ml of sulphuric acid into one litre of water, cool and add 400 ml of nitric acid, dilute to two litres.

5.2.4 Silver Nitrate Solution — 8 g/l.

5.2.5 Hydrofluoric Acid (40 percent)

5.2.6 Ammonium Persulphate — Solid.

5.2.7 Dilute Hydrochloric Acid — 1:1 (v/v).

5.2.8 Standard Ferrous Ammonium Sulphate Solution (0.03 N)

Dissolve 3.2 g of $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ in 500 ml of cold dilute sulphuric acid (5:95) and dilute to one litre with this sulphuric acid. Standardize against re-crystallized potassium dichromate solution.

5.2.9 Standard Potassium Permanganate Solution (0.03 N)

Dissolve 0.958 g of potassium permanganate in one litre of water. Store in a dark coloured stoppered glass bottle. Standardize against pure re-crystallized sodium oxalate or oxalic acid.

5.3 Procedure

5.3.1 Transfer 1 g of accurately weighed sample to a 400 ml beaker. Add 30 ml of the sulphuric acid-nitric acid mixture and 20 ml of silver nitrate solution. Heat gently until the sample is dissolved. If free silicon is present, add few drops of hydrofluoric acid. Boil until brown fumes have been expelled.

5.3.2 Dilute to 300 ml with hot water, add 2 g of ammonium persulphate and heat to boiling. After the chromium is oxidized, boil for 10 min. Add 0.5 ml of dilute hydrochloric acid and continue boiling until any manganese present is reduced. If necessary, add an additional 0.5 ml portions of dilute hydrochloric acid and boil for 15 min after the final addition.

5.3.3 Cool and add a measured volume of ferrous ammonium sulphate solution until colour changes from yellow to green (7 ml of ferrous ammonium

sulphate will reduce about 0.01 g of chromium). Titrate with standard potassium permanganate solution until a faint pink end point is obtained which persists for one minute.

5.4 Calculation

$$\text{Chromium, percent by mass} = \frac{(V_1 N_1 - V_2 N_2) \times 1.734}{m}$$

where

V_1 = volume, in ml of the standard ferrous ammonium sulphate solution used;

N_1 = normality of the standard ferrous ammonium sulphate used;

V_2 = volume, in ml, of the standard potassium permanganate solution used;

N_2 = normality of the standard potassium permanganate solution used; and

m = mass, in g of the sample taken.

Indian Standard

CHEMICAL ANALYSIS OF ALUMINIUM AND ITS ALLOYS

PART 9 DETERMINATION OF TIN

(Second Revision)

1 SCOPE

This standard (Part 9) describes the method for determination of tin by iodimetric method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1967	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF TIN BY IODIMETRIC METHOD

5.1 Outline of the Method

Tin in the sample is completely dissolved in hydrochloric acid in the presence of antimony. Stannous chloride thus formed is titrated with standard potassium iodate solution in carbon dioxide atmosphere.

5.2 Apparatus

A 500 ml conical flask fitted with a three-holed rubber stopper containing an inlet tube for carbon dioxide, an air condenser, and a hole for a burette that shall be kept closed by means of a glass rod, until the titration is started. During the reduction, a very slow stream of carbon dioxide shall be passed through the flask. When

the reduction is complete, the flow shall be increased to maintain a protective cover of carbon dioxide during cooling and titration (*see* Fig. 1).

5.3 Reagents

5.3.1 *Antimony* — Metallic powder.

5.3.2 *Aluminium Sheet or Rolled Foil* — Tin free.

5.3.3 *Dilute Hydrochloric Acid* — 1:2 (v/v).

5.3.4 *Carbon Dioxide* — Gas.

5.3.5 *Starch* — *Potassium Iodide Solution*

Make a paste of 1 g of soluble starch in about 5 ml of water and add to 100 ml of boiling water. Cool and add 40 g of potassium iodide and stir until the potassium iodide is dissolved. Prepare fresh as needed.

5.3.6 *Standard Potassium Iodate Solution* (0.025 N)

Dissolve 0.892 g of potassium iodate (dried at 180°C) in 200 ml of water, containing 1 g of sodium hydroxide and add 10 g of potassium iodide. When solution is complete, dilute to one litre in a volumetric flask. Standardize against tin solution as prepared under 5.3.7.

5.3.7 *Standard Tin Solution*

Dissolve 1 g of tin in a covered 400 ml beaker in 300 ml of dilute hydrochloric acid (1:1) by warming gently until the metal dissolves. If the solution is difficult, add 0.05 to 0.1 g of potassium chlorate. Cool and dilute to one litre in a volumetric flask.

5.4 Procedure

5.4.1 Transfer 2 to 5 g of the accurately weighed sample to a 500 ml flask and add 0.25 g of powdered antimony and 150 ml of dilute hydrochloric acid. After the reaction ceases, heat the flask finally, boiling for about 10 min, to ensure complete solution of tin. Filter and wash with dilute hydrochloric acid. Discard the residue and return the filtrate to the original flask.

5.4.2 Add 0.25 g of powdered antimony and 0.5 g of aluminium sheet or rolled foil free from tin. (If silicon content is low then the addition of aluminium may not be required). Stopper the flask and assemble the

apparatus as shown in Fig.1. Start passing slow current of carbon dioxide gas through the flask. Heat gently until the aluminium is completely dissolved and boil for 10 min. Increase the flow of the gas so as to prevent back pressure and cool. As soon as cold, with the current of carbon dioxide gas still passing, open the stoppered-hole and add from a pipette 5 ml of fresh starch potassium iodide solution. Insert the tip of the burette containing standard potassium iodate solution of suitable strength and titrate to a blue colour.

5.4.3 Carry out a blank determination, following the procedure given in **5.4.1** and **5.4.2** and using the same amounts of all reagents, but without the sample.

5.5 Calculation

$$\text{Tin, percent by mass} = \frac{(A - B)C}{D} \times 100$$

where

A = volume in ml of the standard potassium iodate solution required to titrate the sample,

B = volume in ml of the standard potassium iodate solution required to titrate the blank,

C = tin equivalent of the standard potassium iodate solution in g/ml, and

D = mass in g of the sample taken.

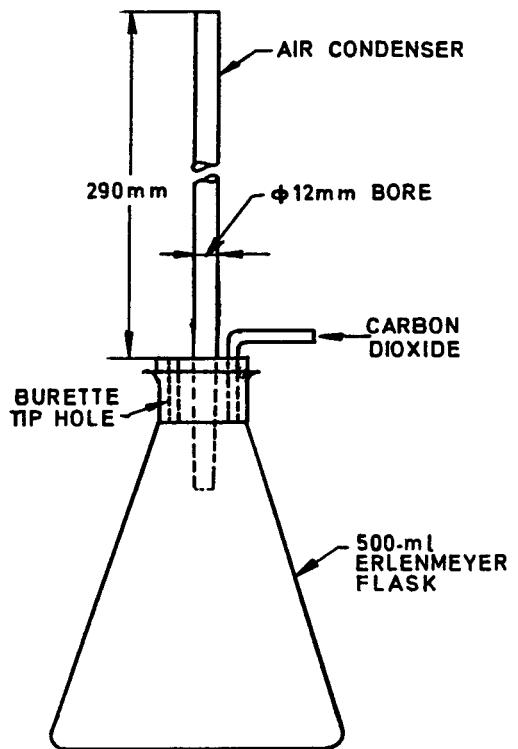


FIG. 1 APPARATUS FOR REDUCTION OF TIN

*Indian Standard***CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS****PART 10 DETERMINATION OF ANTIMONY***(Second Revision)***1 SCOPE**

This standard (Part 10) describes the method for determination of antimony by the potassium iodide-thiourea (photometric) method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

**5 DETERMINATION OF ANTIMONY BY THE
POTASSIUM IODIDE-THIOUREA
(PHOTOMETRIC) METHOD****5.1 Outline of the Method**

In acid solution of the sample, a strong yellow colour is produced by the addition of excess potassium iodide. In the presence of thiourea, copper is kept in solution as a colourless complex.

NOTE — Interference due to bismuth is ignored as this chemical is normally not found in aluminium alloys.

5.2 Reagents

5.2.1 Sodium Hydroxide Solution — 40 percent (v/v).

5.2.2 Dilute Sulphuric Acid — 1:1 (v/v).

5.2.3 Dilute Nitric Acid — 1:1 (v/v).

5.2.4 Hydrazine Sulphate — Solid.

5.2.5 Thiourea — Solid.

5.2.6 Potassium Iodide Solution — 50 percent (m/v).

5.2.7 Standard Antimony Solution

Dissolve 2.003 7 g of potassium antimonious tartrate (dried at 100°C for an hour) in 100 ml of hydrochloric acid and make up to one litre. Alternatively dissolve 0.400 g of pure antimony in aqua regia, add 100 ml of hydrochloric acid and make up to one litre.

NOTE — Check the antimony content by iodimetric titration.

5.3 Procedure

5.3.1 Dissolve 0.40 g of an accurately weighed sample in 10 ml of sodium hydroxide solution. Warm and after the attack is complete, add 15 ml of dilute sulphuric acid followed by 10 ml of dilute nitric acid. Boil, cool for about 1 min, add 0.25 g of hydrazine sulphate to reduce ferric iron to ferrous state. Boil for 2 min, cool to room temperature and transfer to a 200 ml volumetric flask. Add 2 to 3 g of thiourea followed by 40 ml of potassium iodide solution. Make up to the mark and mix.

5.3.2 Take a suitable aliquot, filter through medium textured filter paper, transfer it to an absorption cell and adjust the photometer to the initial setting. Take the spectrophotometric readings using a light filter at approximately 15 min after the addition of thiourea as otherwise turbidity by decomposition of thiourea may occur.

5.3.3 Calibration Curve

Take a series of standard antimony solutions and a reagent blank. Using the same quantities of reagents, carry through all the stages as described under 5.3.1 and 5.3.2 and record the photometric readings of the standard solutions along with the blank. Draw calibration curve by plotting the spectrophotometric readings of standard solutions against milligrams of antimony per 100 ml of the solution.

Convert the spectrophotometric readings of the sample to milligrams of antimony by means of calibration

curve and calculate the percentage of antimony.

5.4 Calculation

$$\text{Antimony, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg, of antimony found in the sample represented by the aliquot solution used; and

B = mass in g, of the sample represented by the aliquot taken.

Indian Standard

**CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS**

PART 11 DETERMINATION OF TITANIUM

(Second Revision)

1 SCOPE

This standard (Part 11) describes the photometric method for determination of titanium, in aluminium and its alloys. It also describes an alternate spectrophotometric method for determination of titanium in the range 0.001 to 1.0 percent using Diantipyrylmethane.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see IS 1070*) shall be employed for the test.

5 DETERMINATION OF TITANIUM BY THE HYDROGEN PEROXIDE (PHOTOMETRIC) METHOD

5.1 Outline of the Method

The sample is subjected to the action of sodium hydroxide and acidified with nitric acid-sulphuric acid mixture. The yellow coloured complex produced in acid solution of the sample by the addition of hydrogen peroxide is determined photometrically at approximately 410 nm.

5.2 Reagents

5.2.1 Sodium Hydroxide Solution — 20 percent (m/v).

5.2.2 Nitric Acid-Sulphuric Acid Mixture

Add 50 ml of nitric acid (r.d. = 1.42) to 600 ml of water, then add 350 ml of sulphuric acid (r.d. = 1.84), slowly, while stirring and cool the mixture.

5.2.3 Dilute Sulphuric Acid — 1:9 (v/v).

5.2.4 Hydrogen Peroxide — 20 volumes.

5.2.5 Standard Titanium Solution (1 ml = 0.001 5 g of Ti)

Fuse 0.25 g of titania in a covered crucible with 15 g of potassium pyrosulphate and dissolve the cold melt in 200 ml of hot dilute sulphuric acid (1:1). Cool to room temperature and dilute to one litre in a volumetric flask.

5.3 Procedure

5.3.1 Transfer one gram of the accurately weighed sample to a 400-ml beaker, cover and add in small instalments 30 ml of sodium hydroxide solution. When the reaction subsides, wash down the cover and sides of the beaker, boil to complete the reaction and cool. Add 50 ml of nitric acid-sulphuric acid mixture. Boil to dissolve salts, expel brown fumes and cool. Transfer to a 100 ml volumetric flask. Dilute to the mark and mix. Filter through a dried fine textured filter paper discarding the first few millilitres of filtrate. Preserve the filtrate.

5.3.2 Transfer a suitable aliquot from 25 ml portion of the solution preserved under 5.3.1 to an absorption cell and adjust the spectrophotometer to the initial setting, using a light filter at approximately 410 nm. To the rest of remaining portion of the filtrate preserved under 5.3.1, add few drops of phosphoric acid and two drops of hydrogen peroxide and mix. While maintaining the spectrophotometer adjustment, take the spectrophotometric reading of a suitable portion of the solution.

5.3.3 Calibration Curve

Take a series of standard titanium solutions and the reagent blank. Using the same quantities of the reagents carry through all the stages as described under 5.3.2. Record the spectrophotometric readings of the standard solutions along with the blank. Draw a calibration curve by plotting the spectrophotometric reading of the standard solutions against milligrams of titanium per 100 ml of the solution.

Convert the spectrophotometric readings of the sample and the blank solutions to milligrams of titanium by means of the calibration curve and calculate the percentage of titanium.

5.4 Calculation

$$\text{Titanium, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

where

- A* = mass in mg, of titanium found; and
B = mass in g, of the sample taken.

6 DETERMINATION OF TITANIUM BY SPECTROPHOTOMETRIC METHOD USING DIANTIPYRYLMETHANE

6.1 Outline of the Method

Titanium in the sample is converted into Ti^{4+} which in the presence of chromotropic acid forms a yellow coloured complex with diantipyrylmethane between the pH range 1.8 to 2.4. Absorbance of the coloured complex is measured at 280 nm.

NOTE — The elements ordinarily present in the aluminium and its alloys do not interfere. Interference of Fe^{3+} is eliminated by reducing it to Fe^{2+} by ascorbic acid in the sample solutions prior to colour development.

6.2 Reagents

6.2.1 Diantipyrylmethane Solution

Dissolve 5 g of diantipyrylmethane in hydrochloric acid (5N) and make up with hydrochloric acid (5N) to 100 ml in a volumetric flask. Store the solution in a polyethylene bottle and use a freshly prepared solution.

6.2.2 Ascorbic Acid Solution (20 g/l)

Dissolve 2 g of ascorbic acid in water and make up to 100 ml.

6.2.3 Copper Sulphate Solution — 1.25 percent (m/v).

Dissolve 1.25 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and make up to 100 ml.

6.2.4 Titanium Standard Solution (1 ml = 0.5 mg Ti)

Dissolve 3.68 g of potassium titanyl oxalate dihydrate [$\text{K}_2\text{TiO}(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$] with 8 g of ammonium sulphate in 100 ml sulphuric acid. Heat to dissolve and quantitatively transfer the contents to one litre volumetric flask, containing about 750 ml water. Cool and make up to volume and mix. Take necessary safety precautions while handling and diluting sulphuric acid.

6.2.4.1 Titanium standard solution (1 ml = 10 μg Ti)

Pipette 10 ml of titanium standard solution (6.2.4) to a 500-ml volumetric flask, dilute to volume and mix.

6.3 Procedure

6.3.1 Weigh accurately 0.250 g of aluminium sample (in the form of drillings or millings) and transfer to a 100 ml beaker, add slowly 5 ml hydrochloric acid (1:1). Heat to dissolve. Wash the sides of the beaker with minimum water. Add 3.75 ml of sulphuric acid (1:1) and mix. Add 1.25 ml of ascorbic acid and mix thoroughly. Add 2-3 drops of copper sulphate solution and 2.5 ml diantipyrylmethane solution and mix.

6.3.2 Transfer quantitatively the contents of the beaker to 25 ml standard flask and make up the volume with water and mix. Wait for 15 min for colour development to complete.

6.3.3 Measure the absorbance of the complex at 380 nm on a spectrophotometer using 10 mm glass cell against reagent blank.

6.3.4 Reagent blank is prepared by following the procedure specified in 6.3.1 (except aluminium sample) to 6.3.3.

6.3.5 Read the titanium concentration from the calibration curve.

6.4 Calibration Curve

6.4.1 Pipette 1,2,3,4,5 and 6 ml of titanium standard solution (6.2.4.1) to a 25-ml standard flask in sequence. Transfer 5 ml hydrochloric acid (1:1) and 3.75 ml sulphuric acid (1:1) and cool. Mix slowly by swirling. Add 1.25 ml of ascorbic acid solution and 2 to 3 drops of copper sulphate solution and mix thoroughly. Add 2.5 ml of diantipyrylmethane reagent solution and mix. Make up the volume with water and mix. Leave the flask for 15 min after colour development. Run the reagent blank.

6.4.2 Measure the absorbance of the coloured complex at 380 nm against reagent blank on a spectrophotometer using 10 mm glass cell. Plot the graph of absorbance against concentration of titanium in μg . This graph is used for finding the titanium concentration in the sample.

6.5 Calculation

$$\text{Titanium, percent by mass} = \frac{M}{m \times 10^4}$$

where

M = mass in μg of titanium found in the aliquot taken; and

m = mass, in g of the sample taken.

*Indian Standard***CHEMICAL ANALYSIS OF
ALUMINIUM AND ITS ALLOYS****PART 12 DETERMINATION OF VANADIUM***(Second Revision)***1 SCOPE**

This standard (Part 12) describes the method for determination of vanadium in the range from 0.005 to 0.02 percent in aluminium and its alloys.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

**5 DETERMINATION OF VANADIUM BY
N-CINNAMOYL-N-PHENYL-HYDROXAMIC
ACID (SPECTROPHOTOMETRIC) METHOD****5.1 Outline of the Method**

The sample is dissolved in dilute hydrochloric acid and is extracted with N-cinnamoyl-N-phenyl-hydroxamic acid in chloroform. The absorbance of the coloured extract is measured at 545 nm using a spectrophotometer.

NOTE — Elements ordinarily present in aluminium metal do not interfere, however, iron interferes during extraction, if present, in high percentage in an alloy.

5.2 Reagents**5.2.1 Potassium Permanganate — 0.5 percent (m/v)**

Dissolve 0.50 g of potassium permanganate in water and make up to volume in 100 ml flask.

5.2.2 N-Cinnamoyl 1-N Phenyl Hydroxylamine (CPHA), 0.5 percent (m/v) in Chloroform

Weigh 0.50 g of N-Cinnamoyl-N-phenyl hydroxylamine in a dry beaker, add 70 ml Chloroform and dissolve the salt. Transfer quantitatively in a dry 100 ml flask with Chloroform and make up to volume with Chloroform.

5.2.3 Dilute Hydrochloric Acid — 1:1 (v/v).**5.2.4 Hydrochloric Acid, r.d.=1:16.****5.2.5 Chloroform****5.2.6 Dilute Sulphuric Acid — 10 percent (v/v).****5.2.7 Standard Vanadium Solution****5.2.7.1 Standard vanadium solution A (500 µg/ml)**

Weigh 0.574 2 g of ammonium metavanadate and dissolve in water (allow to stand overnight to dissolve) transfer quantitatively to a 500 ml standard flask and make up to volume.

Alternatively a little dilute sulphuric acid is added to the salt and the solution is warmed gently to dissolve the solution. Cool and make up to 500 ml in a flask.

5.2.7.2 Standard vanadium solution — B (50 µg/ml)

Pipette out 20 ml of solution A in a 200 ml flask and make up to volume with water and mix well. The solution should be prepared just before use.

5.3 Procedure**5.3.1** Weigh accurately 0.500 g of sample (drillings or turnings) and transfer to a 250 ml beaker. Add 20 ml hydrochloric acid (1:1).

NOTE — If reaction is too vigorous, add a small amount of distilled water down the sides of beaker to moderate the reaction.

5.3.2 When reaction is complete place it on a hot plate and evaporate (not to dryness) until salts crystallize out. Remove from the hot plate and wash down the sides of beaker with 10 to 15 ml of water. Warm the solution on a hot plate to dissolve crystals.

5.3.3 Remove from hot plate. Cool and transfer to 100 ml separating funnel with 5-10 ml of washings. Add potassium permanganate dropwise until a pink colour persists. Add 20 ml of hydrochloric acid and mix well.

5.3.4 Add 10 ml of hydrochloric acid and mix well. Add 20 ml CPHA solution (5.2.2) and mix well. Allow the organic layer to separate and after drying the stem of the funnel, carefully transfer the organic layer to a clean and dry 25 ml standard flask with the help of a small filter funnel. Repeat the extraction several times until the organic layer is colourless and transfer the extract into the same flask.

5.3.5 Prepare a process reagent blank (without sample) and following the procedure as given in 5.3.1 to 5.3.4.

5.3.6 Measure the absorbance of the sample extract in chloroform as obtained in 5.3.4 against a process reagent blank at 545 nm using 10 mm glass cells.

5.3.7 Find the concentration in μg of the extract from the calibration curve.

5.3.8 Calibration Curve

5.3.8.1 Pipette 5.0, 10.0 and 20.0 ml of vanadium solution (5.2.7.2) into three 100-ml flasks and make up to volume with water and mix. Pipette 10 ml solution of these into three different 10-ml separating funnels. Add potassium permanganate solution (0.5 percent) dropwise until pink colour persists.

5.3.8.2 Follow rest of the procedure as specified in 5.3.4.

5.3.8.3 Wash the funnel with 5 ml Chloroform twice and transfer all washings to the same 25-ml standard flask. Wash the funnel with little Chloroform and make up the volume with Chloroform.

5.3.8.4 Prepare a reagent blank by taking 10 ml distilled water (in place of vanadium solution) and following the procedure as given in 5.3.8.1 to 5.3.8.3.

Measure the absorbance of the vanadium extract 5.3.8.3 against the reagents blank obtained in 5.3.8.4 using 10 mm glass cells at 545 nm.

5.3.8.5 Plot the graph of absorbance against concentration of vanadium per 25 ml of chloroform extract. This graph is used for finding the concentration of vanadium in aluminium sample solution.

6 CALCULATION

$$\text{Vanadium, percent by mass} = \frac{M}{m \times 10^6}$$

where

M = mass, in μg of vanadium obtained from the aliquot taken, and

m = mass, in g of the sample taken.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

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This Indian Standard has been developed from Doc : No. MTD 28 (3887).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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